



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:  
Satyavolu, et al.

Serial No. 09/689,994  
Filed: October 13, 2000

Group Art Unit : 1731  
Examiner: J.A. Fortuna

FOR: ENHANCED FIBER ADDITIVE; AND USE

MAIL STOP RCE  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. 1.132

I, Jagannadh Satyavolu, declare that my educational background is as follows:

Ph.D, Chemical Engineering, The Ohio State University, Columbus, Ohio, 1989  
M.S., Chemical Engineering, The Ohio State University, Columbus, Ohio, 1984  
B.Tech., Chemical Engineering, Andhra University, Waltair, India, 1982

Since my graduation, my employment was as follows:

|                        |  |
|------------------------|--|
| 2000-Present           | Cargill, Inc., North American Industrial Starches, Cedar Rapids, IA, Lead Research Engineer  |
| 1992-2000              | Cargill, Inc., Central Research, Process Technology Group, Minneapolis, MN, Senior Research Engineer, August, 1995-2000; Research Engineer, July, 1992 – August, 1995  |
| 1990-1992              | Georgia Institute of Technology, School of Chemical Engineering, Atlanta, GA. Research Scientist II and instructor, Pulp and Paper Eng., April, 1991 to July, 1992; Research Scientist I, Pulp and Paper Eng., February, 1990 to April, 1991 |
| 1986-1990              | Batelle Columbus Labs, Columbus, Ohio; Research Scientist, Process Engineering, March, 1989 to February, 1990; Research Intern, June, 1986 to December, 1988   |
| 1983-1989              | The Ohio State University, Columbus, Ohio, Instructor, Dept. of Mathematics, September, 1985 to March, 1989  |
| 1978-1982<br>(summers) | Gowthami Straw Board Machinery Industries, Rajahmundry, India<br>Plant supervisor, Summer 1982; Engineering Intern, Summers 1978 to 1981   |

I am a co-inventor of the invention described in the above-identified U.S. Patent Application Serial No. 09/689,994.

Under my immediate supervision, Example III of U.S. Patent No. 4,181,747 to Kickle et al. (herein the '747 patent) was reproduced to determine the cellulose content of the product produced in Example III.

Following the procedure of Example II of the '747 patent, 56.1g soy hulls having moisture content of about 10.86% (50g dry weight) were added to 903g of deionized water to provide a slurry containing 5.2% solids. The slurry was brought to a pH of about 4.1 using 23.6g hydrochloric acid (37.6% concentration), and maintained at a temperature of about 71°C to about 76.6°C in a water bath. After 30 minutes at this temperature and pH, the slurry was dewatered. The slurry had a pH of about 4.22 and a 22% solids content. The slurry was reslurried to a solids content of about 4% (163.48g of dewatered solids and 736.52g deionized water), the pH being 4.62. The slurry was agitated for 30 minutes at 60°C, dewatered, and dried to a final moisture not exceeding 10%, with the pH being 4.53. The resulting product and the starting material (the soy hulls) were analyzed for carbohydrate content using the following procedure, referred to as High Performance Anion Exchange Chromatography with Pulsed Amperometric Detection (HPAE-PAD).

#### Sample and Standard Preparation

Each sample was dried and milled to pass through a 40-mesh screen. The moisture of each sample was determined using an NIR moisture balance set at 130°C. Samples were prepared according to TAPPI method T249 cm-85, Tappi Test Methods, Tappi Press, Atlanta, GA, 1985. (The complete disclosure of this Tappi method is incorporated herein by reference). To summarize, 40-60 milligrams of sample was weighed into a glass test tube. To the material in the tube, exactly 1 ml of 72% sulfuric acid was added. The samples were held in a water bath for 1 hr at 30°C, with occasional stirring using a glass stir rod to facilitate dissolution of the sample material. Hydrolyzates were then diluted to 2.5% (w/w) sulfuric acid with deionized water and placed in an autoclave at  $103 \pm 9$  kPa for 60 minutes. After hydrolysis, samples were diluted to 1000 ml in a volumetric flask and filtered through a 0.45 micron nylon syringe filter prior to injection. Standard solutions were hydrolyzed in the same manner as the samples.

### Chromatographic Conditions

Carbohydrates were separated and quantitated using High Performance Anion Exchange Chromatography with Pulsed Amperometric Detection (HPAE-PAD). The DX500 chromatography system (Dionex Corporation, Sunnyvale, CA) consisted of gradient pump (model GP50), an autosampler (model AS-50) equipped with a Rheodyne injection valve, and an electrochemical detector (model ED-40) with a gold working electrode and Ag/AgCl reference electrode. A CarboPac PA-1 analytical column (250 mm x 4 mm i.d.) and guard column (50 mm x 4 mm i.d.) were used to separate the carbohydrates. The pulsed amperometric waveform settings E1, E2, E3 and E4 were set at +0.1, -2.0, +0.6 and -0.1 V for durations of 400, 10, 30 and 60 msec, respectively, for a total of 500 msec, in accord with published Dionex Technical Note 21, incorporated herein by reference. Eluents were prepared using filtered, degassed and deionized high purity water and stored under pressurized Helium. To clean the column, 100 mM NaOH was pumped at 1ml/min for 10 minutes, deionized water was pumped at 1ml/min for 10 min to reequilibrate the column, and the carbohydrates were eluted by pumping deionized water at 1 ml/min for 40 minutes. To stabilize the baseline and optimize detector sensitivity, 300 mM NaOH was added postcolumn at 0.6 ml/min, in accord with, Dionex Technical Note 20, incorporated herein by reference. The total run time per sample was 60 minutes.

### Results

Response factors (RF) for each monosaccharide were determined by dividing the peak area of each carbohydrate by its corresponding concentration. Analyte concentrations are based on the dry weight of the sample material and reported to the nearest 0.1% as the average of two duplicate determinations using external calculation techniques. All concentrations are based on the anhydrous weight equivalent of each carbohydrate, e.g. 0.88 for arabinose and xylose, and 0.90 for galactose, glucose and mannose.

The results are summarized below in the Table.

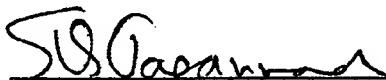
Results – Carbohydrates:

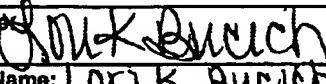
| <u>Carbohydrate</u> | <u>Starting Material<br/>(% dry basis)</u> | <u>Treated Material per Example III<br/>(% dry basis)</u> |
|---------------------|--|---|
| Arabinose           | 4.54                                       | 4.85  |
| Rhamnose            | 0.89                                       | 0.89  |
| Galactose           | 2.72                                       | 2.05  |
| Glucose             | 41.26                                      | 42.4  |
| Xylose              | 8.4  | 9.02  |
| Mannose             | 6.4  | 4.58  |

From the above data, it is shown that the cellulose content of the soy hull starting material and the soy hull product of Example III of the '747 patent are below 50%. The cellulose values are obtained by reference to the glucose content, namely, 41.26% and 42.4%, respectively. Accordingly, the product of Example III of the '747 patent is not a product that is encompassed by the claims of my above-identified U.S. Patent Application Serial No. 09/689,994. As claimed, the products of my application must have a cellulose content of at least 50%.

The undersigned declares further that all statements made herein on his own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with a knowledge that willful, false statements, and the like so made are punishable by fine, or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issues thereon.

August 11, 2004  
Date

  
Jagannadh Satyavolu

| Certificate of Mailing or Transmission   |   |
|--|---|
| I certify that this correspondence is being deposited with the United States Postal Service as First Class Mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, or facsimile transmitted to the U.S. Patent and Trademark Office on the date indicated below: |   |
| Sent to Facsimile No.:   | Signature:  |
| Date: Aug 18, 2004   | Printed Name: Lori K. Burch   |